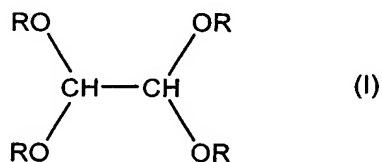


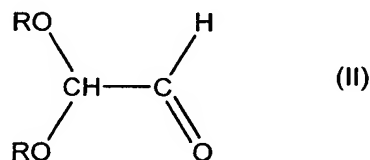
CLAIMS

1. Process for the separation of a glyoxal diacetal of formula (I)



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in which R represents a linear or branched C₁ - C₄ alkyl group, from a crude mixture comprising said glyoxal diacetal and a glyoxal monoacetal of formula (II)



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in which R is as defined above, characterized in that at least one step of countercurrentwise liquid-liquid extraction of said glyoxal diacetal is carried out using a solvent which is immiscible with the reaction medium, in order to obtain, on the one hand, a light phase comprising said glyoxal diacetal and, on the other hand, a heavy phase including the other constituents of the crude mixture.

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2. Process according to Claim 1, characterized in that said crude mixture comprises predominantly a glyoxal diacetal of formula (I) as defined in Claim 1, a glyoxal monoacetal of formula (II) as defined in Claim 1, and water.

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3. Process according to Claim 1 or Claim 2, characterized in that the solvent is chosen from ethers, alkanes and aromatic hydrocarbons.

4. Process according to any one of Claims 1 to 3, characterized in that the solvent is chosen from cyclohexane, n-heptane and toluene.

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5. Process according to any one of Claims 1 to 4, characterized in that the solvent/crude mixture ratio by weight is between 0.3/1 and 5/1.

6. Process according to any one of Claims 1 to 5, characterized in that the extraction is carried out at a temperature of approximately 10°C to 60°C, preferably at ambient temperature.

5 7. Process according to any one of Claims 1 to 6, characterized in that the light phase comprising the glyoxal diacetal of formula (I) and the solvent is subjected to a separation, on conclusion of which said glyoxal diacetal is recovered.

8. Process according to Claim 7, characterized in that this separation is carried out by distillation under reduced pressure.

10 9. Process according to either one of Claims 7 and 8, characterized in that this separation is carried out at a temperature of between ambient temperature and approximately 120°C.

10. Process according to any one of Claims 1 to 9, characterized in that the solvent is recycled to the liquid-liquid extraction step.

15 11. Process according to any one of Claims 1 to 10, characterized in that the crude mixture is obtained by an acetalization reaction of 40 to 75% by weight aqueous glyoxal with an alcohol of formula R-OH in which R is as defined in Claim 1, the R-OH/glyoxal molar ratio being between 10/1 and 50/1, preferably 10/1 to 30/1, in the presence of an acid catalyst, followed by
20 the distillation of the reaction mixture obtained in order to remove the excess alcohol R-OH.

12. Process according to any one of Claims 1 to 11, characterized in that, in the formulae (I) and (II), R is a C₁-C₂ alkyl group.

25 13. Process according to Claim 12, characterized in that R is a methyl group.

14. Process according to any one of Claims 1 to 13, characterized in that the alcohol is methanol.

30 15. Process according to any one of Claims 1 to 14, characterized in that the crude mixture comprises predominantly 1,1,2,2-tetramethoxyethane (TME), dimethoxyethanal (DME) and water.

16. Process according to any one of Claims 1 to 15, characterized in that said mixture comprises, as percentages by weight, approximately 25 to 60% of TME, approximately 7 to 35% of DME and approximately 20 to 50% of water.

5 17. Process according to any one of Claims 1 to 16, characterized in that said mixture also comprises, as percentages by weight, approximately 0 to 15% of glyoxal, approximately 0 to 10% of methanol and approximately 0 to 5% of impurities.

10 18. Process according to any one of Claims 11 to 17, characterized in that the glyoxal used in the acetalization reaction is concentrated to approximately 60 to 70%.

19. Process according to Claim 18, characterized in that the glyoxal is concentrated from an aqueous solution.

15 20. Process according to any one of Claims 11 to 19, characterized in that the acetalization reaction is carried out for a period of time of less than or equal to 1 h, preferably of less than or equal to 40 min.

21. Process according to Claim 20, characterized in that the period of time of the reaction is less than or equal to 20 min.

20 22. Process according to one of Claims 11 to 21, characterized in that the acetalization reaction is carried out at a temperature of the order of 60°C to 140°C, preferably approximately 80°C to 130°C.

23. Process according to Claim 22, characterized in that the temperature is of the order of 100 to 130°C.

25 24. Process according to one of Claims 11 to 23, characterized in that the acetalization reaction is carried out at a pressure of greater than or equal to atmospheric pressure.

25. Process according to Claim 24, characterized in that the pressure is less than or equal to 15 bar.

30 26. Process according to any one of Claims 1 to 25, characterized in that the acetalization reaction, the liquid-liquid extraction step and the

recovery of the various constituents of the crude mixture are carried out continuously, the glyoxal, the glyoxal monoacetal, the alcohol R-OH and the extraction solvent being recycled.